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ACADEMICIAN SERGEY SEMENOVICH NAMETKIN

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 A. S. Nekrasov, N. N. Mel'nikov

[The following are extracts from the above article, which is devoted to the work of the late S. S. Nametkin (1876 - 1950). Only the sections which appeared to be of particular interest were selected. The portion of the article not included is chiefly a review of Nametkin's work on terpenes and essential oils.]

I. NITRATION OF HYDROCARBONS

Academician S. S. Nametkin, on the basis of his work on a thesis at Moscow State University on the chemical reactions and properties of hydrocarbons of Caucasus petroleum (1902), arrived at definite conclusions as to the nature of naphthenes of this petroleum. Wishing to confirm these views, but unable to use catalytic dehydrogenation, which was unknown at that time, he resorted to the nitration of separate petroleum fractions, according to the reaction of M. I. Konovalov. Results of this work, undertaken during the period 1907 - 1911, were combined in Nametkin's Master's dissertation, "Problem of the Action of Nitric Acid on Hydrocarbons of a Saturated Character" (Moscow, 1911), at St Petersburg University.

Not only did Nametkin investigate the action of nitric acid on the simplest alicyclic hydrocarbons, which had been insufficiently studied, and prepare in the pure form and characterize their nitro, amino, and other derivatives but more important, the central interest of these investigations was to clarify the mechanism of nitric acid's action on saturated hydrocarbons.

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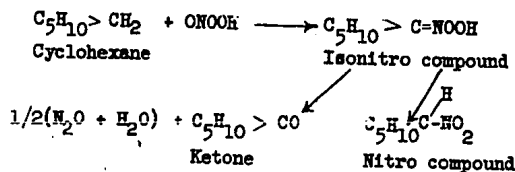
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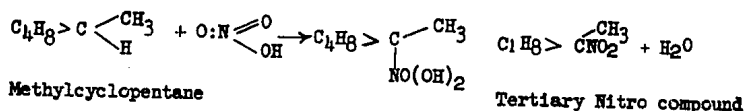
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Nametkin proposed that the primary products of the interaction of nitric acid with saturated hydrocarbons must be not nitro compounds but their desmotropic, unstable forms, the so-called isonitro compounds. The latter immediately undergo conversion, partly by isomerization into stable nitro compounds and partly by decomposition (according to Neff's scheme for the decomposition of isonitro compounds in acidic solution) to form nitrous oxide and a ketone or aldehyde; these ketones and aldehydes are further oxidized by an excess of nitric acid to the corresponding acids. Thus, the interaction of cyclohexane with nitric acid can be represented by the following scheme:



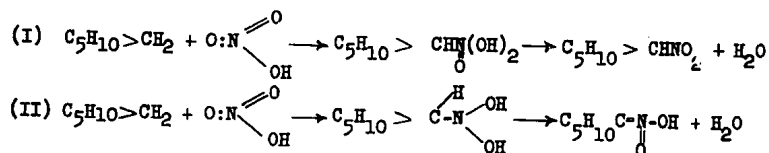
This structural scheme was verified experimentally by Nametkin.

Much later, working in conjunction with A. S. Zabrodina (1925), Nametkin again returned to the problem of the mechanism of nitration of saturated hydrocarbons. Maintaining his earlier viewpoint on the role of isonitro compounds in this reaction, Nametkin this time focused his attention on the reaction's first phase. Proceeding from the assumption that in this case, as in any other substitution reaction, there must be an addition reaction, he took the position that the first interaction product must be, in this case, the product of addition of the hydrocarbon to nitric acid. If a tertiary hydrogen participates in the reaction, then, immediately following the formation of such an addition product, water is split off and the tertiary nitro compound is formed:



The formation of an aromatic nitro compound can be represented by the same scheme.

If, on the other hand, the interaction of nitric acid with the hydrocarbon takes place at its secondary (or primary) hydrogen, the splitting off of water also takes place after formation of the addition product, but in this case, it can proceed in two different directions, as is evident from the following two schemes:



According to scheme (I), the secondary (or primary) nitro compound is formed directly from the initial product of addition of the hydrocarbon to nitric acid; this scheme is clearly analogous to the above-cited scheme of formation of tertiary (or aromatic) nitro compounds. Scheme (II) leads to the formation of the isonitro compound which either is isomerized into the

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stable nitro compound form or decomposes to form nitrous oxide and the ketone (or aldehyde).

In more recent years, Nametkin became drawn to the study of the action of weak nitric acid on saturated hydrocarbons. In particular, he made a detailed study, in conjunction with K. S. Zabrodina, on the nitration of isooctane with weak nitric acid (cf 00-W-17662 and 00-W-17128).

II. PETROLEUM CHEMISTRY

Occupying a special place in the experimental work of Nametkin are the broad investigations on a wide variety of topics in the field of the chemistry and chemical technology of petroleum, which were systematically developed in connection with his work at the State Research Petroleum Institute, starting in 1926, and then, from 1934 on, at the Institute of Mineral Fuels and the Institute of Petroleum, both of the Academy of Sciences USSR.

Among these investigations were a number devoted to the characteristics of petroleum from new, insufficiently studied occurrences. For example, with the participation of L. N. Abakumovskaya, S. S. Nifontova, V. G. Putsillo, S. P. Uspenskiy, and Ye. M. Shakhnazarova, investigations were undertaken on petroleum from Sakhalin and Kamchatka, as well as the first petroleum from the Second Baku; at this point mention can be made of the investigation of asphalt from the great Okhinsk asphalt lake. These works were published from 1925 - 1950.

At the same time, Nametkin conducted investigations on the characteristics of the approximate composition of natural gases and certain petroleum products. Work on combustible natural gases was carried out under the leadership of Nametkin in closest association with A. S. Zabrodina, A. S. Karkanas, D. N. Kurzanova, V. A. Sokolova, S. P. Uspenskiy, and others. The gases selected and analyzed were from the following areas: Baku (nine occurrences), Dagestan (3 occurrences), Chusovoy (Chusovskiye Gorodki occurrence), Melitopol' (2), and Taman' (11). Determination of the composition of the hydrocarbon part of the gases was carried out by distilling them in high vacuum at low temperatures, starting with the temperature of liquid air, in original, specially constructed equipment, over mercury. This was essentially the first such investigation of combustible natural gases in the USSR undertaken on such a large scale (1931 - 1933).

Nametkin's attention was also attracted to the study of the approximate composition of oil fractions of petroleum, solid petroleum products, paraffin waxes, and ceresins (1928 - 1936). To solve the extremely complex problem of the composition of oil fractions of petroleum, Nametkin, in conjunction with Ye. S. Pokrovskaya, began to accumulate synthetic material which could serve for comparison with different oil fractions. This immense, laborious task, which is also being undertaken at a number of foreign laboratories, is now being continued by Pokrovskaya alone.

Extraordinarily interesting results were obtained by S. S. Nametkin working with S. S. Nifontova in the comparative investigation of the approximate composition of different paraffin waxes and ceresin. Lignite paraffin wax, petroleum paraffin wax, and ceresin were subjected to nitration after being carefully purified of oil admixtures. It was shown that the nitration of lignite paraffin wax yields principally the secondary nitro compound of composition $C_{26}H_{53}NO_2$, and no more than 5% of the tertiary nitro compound,

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which is apparently only a by-product. It is therefore clear that lignite paraffin wax consists basically of methane series hydrocarbons of normal structure. Nitration of petroleum paraffin wax also yielded a secondary nitro compound as the chief reaction product, but in this case, with a slightly different composition; in this case, however, considerable quantity (up to 35%) of the tertiary nitro compound of the same composition was obtained. Thus, petroleum paraffin wax contains not only normal hydrocarbons of the methane series but also a considerable quantity of isoparaffins. Finally, ceresin (average molecular composition $C_{45}H_{92}$) yielded, when nitrated, the tertiary nitro compound of composition $C_{45}H_{91}NO_2$ as the chief product; thus, ceresin consists of isoparaffins of the methane series.

By his work in studying the chemical composition of different petroleum, Nametkin obtained essentially the first data which made it possible to estimate the approximate nature of hydrocarbons in different paraffin waxes and ceresin. Naturally, the methods of determining the chemical composition of petroleum products, which, as is well known, are frequently extremely inadequate also attracted Nametkin's attention. The development of convenient methods for the determination of unsaturated and aromatic hydrocarbons in cracking gasoline posed a great deal of difficulty, but as a result of extensive, time-consuming work in conjunction with Ye. A. Robinson, this complex problem was finally solved satisfactorily. Methods were worked out for volumetric determination of unsaturated and aromatic hydrocarbons in one test sample in the following manner: unsaturated hydrocarbons are determined and separated with the aid of sulfur monochloride (S_2Cl_2). If this is done in the cold, aromatics, as has been demonstrated, are in no way affected; aromatic compounds are determined in the residue left after separation of unsaturated hydrocarbons by the usual method (with an excess of concentrated sulfuric acid or the Kattwinkel reagent). In its simplicity of execution and accuracy of results, the method is considered fully reliable and has become widely used.

The method of group analysis of oils was also examined intensively by Nametkin, together with Ye. A. Robinson. However, this extremely complex problem can not yet be considered completely solved. Great difficulties have been encountered in working out methods for the group analysis of sulfur compounds of petroleum. Nametkin's careful investigation in conjunction with V. G. Putsillo and Ye. P. Shcheglova showed that the overwhelming majority of methodical procedures directed toward the separation of individual types of sulfur compounds do not yield very accurate results and require very substantial corrections.

As to the quality of petroleum products, so important to the USSR petroleum industry, Nametkin worked with G. V. Andreyev, V. T. Arkhangel'skiy, A. S. Velikovskiy, S. S. Nifontova, and others to conduct comparative investigations of USSR and foreign petroleum products: gasolines, kerosenes, oils, and waxes. This great experimental work, separate parts of which were published, when completed, in the periodical *Neftyanoye Khozyaystvo*, receiving attention also in the foreign technical press, was published in 1930 in monograph form. There is no doubt that in its time it had a beneficial influence in increasing the quality of production of the USSR petroleum-processing industry.

In the field of the processing of petroleum and petroleum products, Nametkin was principally interested in problems of cracking, especially oxidation cracking according to Dubrovay, as well as problems of aromatization and aromatic cyclization of petroleum fractions. Indeed, Nametkin, in conjunction with engineer S. S. Katsurov and others, took part in the development of the first USSR experimental cracking equipment. In the field of oxidation cracking, he worked with L. M. Rozenberg to study the chemistry of this very

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promising process as it pertained to individual hydrocarbons of different series. In the field of aromatization and aromatic cyclization, again working with Rozenberg, he developed a method for preparation of an extremely active catalyst and successfully studied the process as applied both to individual hydrocarbons of different series and to numerous petroleum products and their fractions.

Among the many problems involved in the purification of petroleum products which are encountered in the experimental investigations of Nametkin, are those of the sulfuric acid purification of different petroleum products, their desulfuration, and increasing their quality by the use of admixtures. Nametkin was interested in sulfuric acid purification chiefly from the viewpoint that a correct understanding of its theoretical basis would aid in rationalizing the purification of petroleum products by this oldest method. Having realized for example, that ceresin consists primarily of isoparaffins which must react with fuming sulfuric acid, Nametkin, working with ~~an~~ R. S. Teneva, demonstrated that the purification of crude ceresin with an excess of this strong reagent must result in high losses; he therefore insisted on a sharp reduction of the quantity of acid employed in this purification process.

More recent observations of R. S. Teneva and S. S. Nifontova revealed that it was possible to dispense entirely with fuming sulfuric acid. This led to the processing of raw ceresin with clays, under which conditions the yields of the final product have increased considerably. Studying the purification of cracking gasoline with sulfuric acid, Nametkin concluded that for the most accurate analysis of the chemical reactions taking place, it was necessary to study in detail the action of strong sulfuric acid on individual hydrocarbons rather than on complex mixtures. Experiments along this line in conjunction with L. N. Abakumovskaya, and later with L. I. Strel'chunas, led to the discovery of a new type of conversion of unsaturated hydrocarbons: hydro- and dehydropolymerization. Essentially, these conversions proceed as follows:

Under the action of strong sulfuric acid, unsaturated hydrocarbons undergo two basic reactions, polymerization and reduction-oxidation; the products of these conversions are, on the one hand, hydrodimers, hydrotrimers, and hydro-polymers in general, and, on the other hand, highly unsaturated compounds bound to sulfuric acid which, on dilution of sulfuric acid with water, are separated in the free state and rapidly polymerized, being converted into resinous and asphaltic dehydropolymers. In further investigations (together with M. G. Rudenko), analogous conversions were observed under the action of aluminum chloride on unsaturated hydrocarbons, but with the difference in this case that the process is complicated by partial destruction, resulting in a much more complex mixture of final reaction products.

The discovery of the Second Baku, whose petroleum is high in sulfur content, posed the problem of desulfurization to the USSR petroleum industry. Nametkin took an active part in solving this problem, conducting with notable success experiments on the purification of certain petroleum products with a high sulfur content with aluminum chloride (together with M. A. Kazarnovskaya), and nitric acid (together with A. S. Sosnina). It is true that these methods received no practical application, but the results obtained by the nitric-acid method enabled Nametkin to reach the important conclusion that according to their chemical properties, sulfur compounds from the kerosene distillate of Chusovskiy Gorodki petroleum are basically thiophenes. With special care, Nametkin, together with D. P. Sanin, S. V. Makover, and A. N. Isyb, studied the desulfurization of high-sulfur shale gasolines by the hydrogenation method.

The experiments were conducted in the presence of a sulfur-resistant catalyst (molybdenum disulfide) in rotating autoclaves or with agitation

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at 350° C and an operative pressure of about 100 atm. By use of such methods, gasoline containing up to 10.6% sulfur was fully desulfurized. At the time (1934), these were the first successful attempts in the USSR to desulfurize totally liquid fuel with such a high sulfur content.

The use of additives is, at present, one of the most efficient means of improving quality and is widely used for both motor fuel and lubricating oils. Nametkin and his associates worked long and unremittingly in this exceptionally important field. In cooperation with P. I. Sanin, S. S. Nifontova, and V. G. Putsillo, a great deal of work was done on the synthesis of depressors (additives lowering the congelation temperature of oils).

Experimental testing of different, newly synthesized depressant admixtures demonstrated that their activity depends very little on the structure or composition of their molecules; a definite dependence, however, was observed between their activity and their molecular weights, or more specifically, the weights of their active groups (radicals), and particularly on the nature of the lubricating oil. A variety of material showed that depressant activity is manifested only in distillate oils, while no activity at all appears in residual oils, a phenomenon which has been observed by other investigators.

Viscosity additives, which raise the viscosity index of an oil when the temperature is elevated, have taken on great importance in the practical employment of lubricating oils. The US additive paratone, a study of which was undertaken a number of years before World War II, with the participation of M. G. Rudenko, has been considered the best additive of this type.

Developing this work further, Rudenko, in association with V. N. Gromova, even before World War II, synthesized the additive superol, which, according to its tests, not only equals but in some respects surpasses the US additive. Most recently, during World War II, Nametkin, in conjunction with P. I. Sanin and N. S. Nametkin, carried out extensive work in the field of scorching additives. Furthermore, Nametkin is responsible for the first scientific and technical classification and systematic survey of additives, which are published in his Khimiya Nefti (Petroleum Chemistry).

As far as such basic products of the modern petroleum industry as motor fuel and various types of lubricating oils are concerned, their production, at present, is continuously taking on more features characteristic of chemical processing of petroleum raw materials. Moreover, the last 15-20 years have seen the development of a number of new methods for the deep chemical processing of petroleum products, which have created a new branch of the petroleum industry, the petroleum-chemical industry. The production of this industry comprises aromatic hydrocarbons, alcohols and phenols, aldehydes and ketones, organic acids, their derivatives, and numerous other products with a variety of practical applications. An ardent supporter of the intensive development of the petroleum-chemical industry in the USSR, Nametkin and his associates have taken an energetic, active part in the development of different methods for the deep chemical processing of petroleum and its products.

In association with D. M. Rozenberg, Nametkin worked long and persistently on the problem of obtaining the simplest aromatics (benzene, toluene, and xylene) as raw materials for the chemical industry by catalytic cyclization and aromatization of narrow petroleum fractions. The chemotechnological basis for this work is the same as for the above-noted aromatization of gasolines.

At present, isobutene is one of the scarcest of the olefins used on an industrial scale. It is required as a raw material for the production of 100-octane gasoline (isooctane) and additives of the isobutylbenzene type.

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for the synthesis of extremely valuable additives to lubricating oils, for the isobutylphenol, etc. Nametkin spent considerable time and labor on the development of methods for increasing the supply of isobutene by the catalytic isomerization of cracking butenes into isobutene and the separation of pure isobutene from the mixture of gases thus obtained. With the help of A. S. Nekrasov, V. G. Putsillo, M. A. Kazarnovskaya, A. S. Sosnina, and P. G. Chernova, this problem was completely solved.

Production of amyl alcohols on a base of petroleum pentene is one of the most highly developed processes of the US petroleum-chemical industry. Nametkin and A. G. Serebrennikova worked out in detail both of the basic stages of this process: continuous chlorination of petroleum pentane, and subsequent conversion of the mixture of chlor-pentanes into amyl alcohols and their acetic esters.

Nametkin, V. K. Zvorykina, and R. S. Teneva showed that oxidation of paraffin wax or gas with oxygen from the air yields fatty acids, the desired products of the process, as well as considerable quantities of alcohols and aldehydes of the same series, from the lower (liquids) to the higher (crystalline). Some of these alcohols and aldehydes, as well as mixtures of them, which until now have been waste products of fatty-acid production by the above method, are of great interest and value to different types of industry; industrial production of this type can be set up in such a manner as to utilize waste products, and this is one of the tasks at hand for the branches of industry which are interested in this problem.

At the suggestion of the Main Administration of the Perfume Industry, Nametkin conducted investigations to find a substitute for soap made from ordinary fat. Working with A. S. Zabrodina, A. S. Kursanova, V. A. Khokhryakova, and others, he found that on the base of petroleum kerosene, it is possible to produce detergents which, added to fatty soap in the amount of 40-50%, not only do not reduce the soap's quality, but in some respects improve it.

III. ORGANOMETALLIC COMPOUNDS

Passing over the numerous works of S. S. Nametkin on the use of organic magnesium compounds for the synthesis of the widest variety of substances, we will only touch on investigations of properties and methods for preparation of organometallic compounds as such.

In the first place, it must be noted that at one time, it fell on the shoulders of Nametkin to prepare in the laboratory enough tetraethyl lead to permit motor testing in the USSR of this most important antiknock compound. With the assistance of D. N. Abakumovskaya and D. N. Kursanov, this responsible and dangerous job was successfully completed, making possible the first testing of USSR gasolines with a TEL additive. Later, working with K. P. Lavrovskiy, Nametkin participated in the development of the technical method for producing tetraethyl lead and in a number of other investigations in the field of motor fuel antiknock agents.

Investigation, in association with V. V. Nekrasov, of the reaction of hydrogen sulfide with certain alkyl- and aryl-dichloroarsines showed that this reaction proceeds quite rapidly, according to the scheme: $\text{RAsCl}_2 + \text{H}_2\text{S} \longrightarrow \text{RAsS} + 2\text{HCl}$. It was shown that when the reaction is conducted in aqueous solution at room temperature, the sulfide, insoluble in water, is precipitated. Thus hydrogen sulfide was proposed as group reagent for organic arsenic compounds of the general formula RAsCl_2 .

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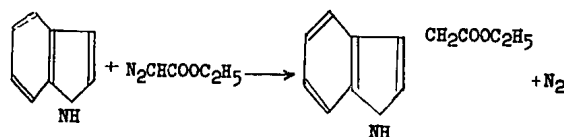
In the same investigation, Nametkin was able to demonstrate that alkyl-dichloroarsines reduce mercury from mercurous nitrate at different rates, depending on the character and molecular weight of the hydrocarbon radical which is bound to arsenic. This observation made it possible for this reaction to be proposed for qualitative differentiation of methyl-, ethyl-, and β -chlorovinyl-dichloroarsines.

To study and employ the A. N. Nesmeyanov reaction for the synthesis of organic thallium compounds, Nametkin and N. N. Mel'nikov synthesized a number of complex compounds, not described in the literature, of thallium chloride with diazonium salts. In the process of the investigation it was established that diazonium salts form with thallium chloride two types of salts, $(\text{ArN}_2)_2\text{TlCl}_5$ and $\text{ArN}_2\text{TlCl}_4$. Special experiments showed that a complex cation, containing thallium and the hydrocarbon radical, enters into the composition of these complexes. These compounds are comparatively unstable, decomposing easily on heating, according to the scheme: $\text{ArN}_2\text{TlCl}_4 \rightarrow \text{ArCl} + \text{TlCl}_3 + \text{N}_2$. Nametkin, N. N. Mel'nikov, and G. P. Gracheva synthesized and described a number of complex organic thallium compounds of the general formulae ArTlHal_2 and Ar_2TlHal . Finally, Nametkin, together with N. N. Mel'nikov, developed a simple method for determining thallium in organic compounds.

IV. PLANT GROWTH STIMULANTS AND HERBICIDES

Nametkin also carried out research in the field of plant-growth stimulants. Immediately on discovery of these agents and determination of the structure of auxins, Nametkin recognized their importance to the national economy and organized not only the synthesis of compounds already known but also the search for new active substances. The study of plant-growth stimulants held his attention during all the last years of his life, his work having been conducted in the laboratory of Moscow State University and at the Laboratory of Chemistry of the Institute of Plant Physiology (Imeni K. A. Tiryazev, Academy of Sciences USSR, which latter laboratory he organized). Nametkin's organization of work on the preparation and study of plant-growth stimulants made possible a wide range of research on the action of these substances on plants, laying the ground for their practical use in plant growing.

Nametkin's work in the field of synthesis of plant-growth stimulants was directed principally toward the study of means for preparing heteroauxin, β -indolyl- γ -butyric acid, α -naphthylacetic acid, and certain very important phenoxyacetic acids and their derivatives. Systematic study of methods for preparing the important stimulant of root formation, heteroauxin, was carried out with the assistance of N. A. Dzbanovskiy and N. A. Favorskaya. This work succeeded in greatly perfecting the preparation of heteroauxin, starting from itaconic acid. The first larger batches of the substance were prepared under laboratory conditions by this method. Satisfactory results were also obtained by experimenting on the preparation of heteroauxin, starting from magnesium indole and chloroacetonitrile, while even better results were arrived at by the reaction of indole with diazoacetic ester:



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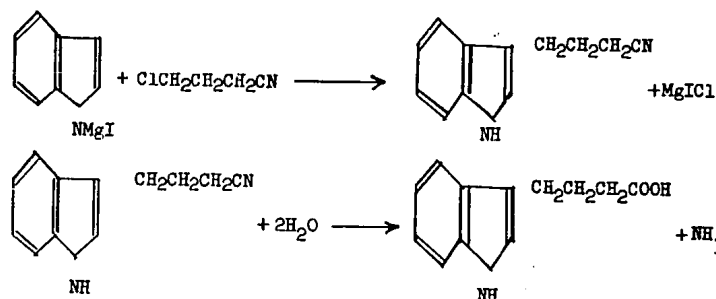
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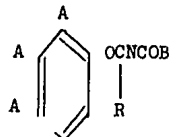
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Although this reaction has long been known, its practical application has been hindered by the impossibility of controlling the decomposition of diazoacetic ester, which sometimes proceeds extremely violently. But Nametkin, N. N. Mel'nikov, and K. S. Bokarev succeeded in finding conditions which permit the reaction rate to be regulated and the reaction to be used for preparation of large quantities of heteroauxin.

Working with N. A. Dzbanovskiy and A. G. Rudnev, Nametkin worked out a new method for preparation of β -indolyl- γ -butyric acid, which is an even more active stimulant of root formation than heteroauxin. This reaction, which is based on the reaction of γ -chlorobutyronitrile with magnesium iodoindole, can be represented by the following schemes:

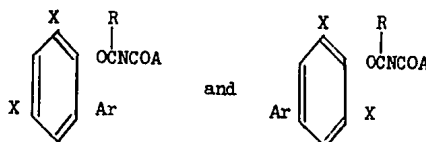


Phenoxyalkylcarboxylic acids occupy an important place in Nametkin's works on plant-growth stimulants. Nametkin, N. N. Mel'nikov, and K. S. Bokarev proposed a large group of substances of the following general formula:



where A is an alkoxyl radical or hydrogen, R is an aliphatic hydrocarbon radical or hydrogen, and B is a hydroxyl, amido group, or arylamido group.

Nametkin, N. N. Mel'nikov, and Yu. A. Baskakov also proposed as plant-growth stimulants compounds of the following general formulae:

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where X is a halogen, R an aliphatic hydrocarbon radical or hydrogen, Ar an aromatic hydrocarbon radical, CH_3 , C_2H_5 , or C_6H_5 , and A the hydroxyl, amido group, or arylamido group.

Nesetkin, Mel'nikov, Baskalov also described a series of halogen-naphthoxyacetic and halogen-naphthoxybutyric acids, in which groups they synthesized more than 200 compounds not described in the literature. This work resulted in the recommendation of a number of substances for practical application, both as plant-growth stimulants and as herbicides.

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